Institute for
Interlaboratory Studies

Results of Proficiency Test
Total Metals (by digestion)
in Textile
November 2023

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since the 1990's many countries have adopted environmental standards and requirements restricting the use of harmful chemicals in the production of textiles and clothing. Laws and regulations impose some of these standards and requirements. In addition to mandatory environmental standards and requirements for textiles some Ecolabelling schemes are imposing environmental requirements for textile products on a voluntary basis e.g. EU Ecolabel regulation 2014/350/EU, OEKO-TEX® Standard (Switzerland), BlueSign® (Switzerland) and American Apparel and Footwear Association (United States).

On request of a number of participants the Institute of Interlaboratory Studies (iis) decided to organize a proficiency scheme for the determination of Total Metals (by digestion) in Textile.

In this interlaboratory study 70 laboratories in 22 countries registered for participation, see appendix 4 for the number of participants per country. In this report the results of Total Metals (by digestion) in Textile proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to a laboratory that has performed the tests in accordance with for ISO/IEC17043 relevant requirements of ISO/IEC17025.
It was decided to send one textile sample of approximately 3 grams labelled #23745.
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

A batch of red cotton was selected, which was artificially fortified with some elements by a third party. After homogenization 80 small plastic bags were filled with approximately 3 grams each and labelled #23745.
The homogeneity of the subsamples was checked by determination of Copper and Lead according to EPA 3052 on 8 stratified randomly selected subsamples.

	Copper as Cu in mg/kg	Lead as Pb in mg/kg
sample #23745-1	78.13 G(0.05)	281.66
sample #23745-2	69.42	263.16
sample #23745-3	66.11	281.45
sample #23745-4	67.08	279.80
sample #23745-5	64.08	271.66
sample #23745-6	64.42	279.81
sample #23745-7	62.96	257.30
sample #23745-8	60.44	255.65

Table 1: homogeneity test results of subsamples #23745

Subsample 1 is for Copper a Grubbs outlier and therefore for Copper excluded from statistical evaluation of the homogeneity.

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibility of the reference test methods in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Copper as Cu in mg/kg	Lead as Pb in mg/kg
r (observed)	8.17	31.08
reference test method	EN16711-1:15	EN16711-1:15
0.3 x R (reference test method)	7.64	41.02

Table 2: evaluation of the repeatabilities of subsamples #23745

The calculated repeatabilities are in agreement with 0.3 times the corresponding reproducibility of the reference test methods. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories a textile sample labelled #23745 was sent on October 4, 2023.

2.5 ANALYZES

The participants were requested to determine total content of the elements Antimony as Sb, Arsenic as As, Cadmium as Cd, Chromium as Cr, Cobalt as Co, Copper as Cu, Lead as Pb, Mercury as Hg and Nickel as Ni.
It was requested not to use less than 0.5 gram per determination. It was also requested to report if the laboratory was accredited for the determined components and to report some analytical details.

It was explicitly requested to treat the sample as if it was a routine sample and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report ‘less than’ test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per determination in appendices 1 and 2 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the result tables in appendices 1 and 2. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report ‘iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation’ of June 2018 (iis-protocol, version 3.5).
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as ‘<…’ or ‘>…’ were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either ‘unknown’, ‘OK’, ‘suspect’ or ‘not OK’. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner’s outlier test can be used. Outliers are marked by D(0.01) for the Dixon’s test, by G(0.01) or DG(0.01) for the Grubbs’ test and by R(0.01) for the Rosner’s test. Stragglers are marked by D(0.05) for the Dixon’s test, by G(0.05) or DG(0.05) for the Grubbs’ test and by R(0.05) for the Rosner’s test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT the criterion of ISO13528, paragraph 9.2.1, was met for all evaluated tests. Therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements (derived from e.g. ISO or ASTM test methods), the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

$z_{(target)}$ = (test result - average of PT) / target standard deviation

The $z_{(target)}$ scores are listed in the test result tables in appendix 1.

Absolute values for z<2 are very common and absolute values for z>3 are very rare. Therefore, the usual interpretation of z-scores is as follows:

	|z|	< 1	good
1 <	|z|	< 2	satisfactory
2 <	|z|	< 3	questionable
3 <	|z|		unsatisfactory

4 EVALUATION

In this proficiency test no problems were encountered with the dispatch of the samples. Three participants reported test results after the final reporting date and three other participants did not report any test results. Not all participants were able to report all tests requested.
In total 67 participants reported 190 numerical test results. Observed were 5 outlying test results, which is 2.6%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

Not all data sets proved to have a normal Gaussian distribution. These are referred to as “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1 EVALUATION PER ELEMENT

In this section the reported test results are discussed per element. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 5.

For the determination of Total Metals by digestion in Textile the EN16711-1 is considered to be the official test method. This method mentions the standard deviation and variation coefficient per element between laboratories. The reproducibility of each metal was calculated by multiplying the variation coefficient of the metal with 2.8.

Cadmium as Cd, total: The group of participants met the target requirements. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN16711-1:15.

Copper as Cu, total: The group of participants met the target requirements. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of EN16711-1:15.

Lead as Pb, total: The group of participants met the target requirements. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of EN16711-1:15.

The participants agreed on a concentration near or below the limit of detection for all other elements mentioned in paragraph 2.5. Therefore, no z-scores are calculated for these elements. The reported test results are given in appendix 2.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from reference methods are presented in the next table.

Element	unit	n	average	2.8 * sd	R(lit)
Cadmium as Cd, total	mg/kg	62	53.12	10.18	11.90
Copper as Cu, total	mg/kg	57	51.52	10.42	20.20
Lead as Pb, total	mg/kg	66	250.1	53.9	126.1

Table 3: reproducibilities of test on sample #23745

Without further statistical calculations it can be concluded that for all tests there is a good compliance of the group of participants with the reference test method.

4.3 PERFORMANCE OF THE PROFICIENCY TEST OF NOVEMBER 2023

	November 2023
Number of reporting laboratories	67
Number of test results	190
Number of statistical outliers	5
Percentage of statistical outliers	2.6%

Table 4: overview of the proficiency test

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations of the proficiency test expressed as relative standard deviation (RSD) of the PT, is shown in the next table.

Element	November 2023
Antimony as Sb, total	--
Arsenic as As, total	--
Cadmium as Cd, total	7%
Chromium as Cr, total	--
Cobalt as Co, total	--
Copper as Cu, total	7%
Lead as Pb, total	8%
Mercury as Hg, total	--
Nickel as Ni, total	--

Table 5: the uncertainty of the PT

4.4 EVALUATION OF THE ANALYTICAL DETAILS

For this PT some analytical details were requested and are listed in appendix 3. Based on the answers given by the participants the following can be summarized:

- 94% of the participants mentioned that they are ISO/IEC17025 accredited to determine the reported elements.
- 36% of the participants mentioned to use the sample as received and 64% did further cut or further grind the sample prior to analysis.
- The sample intake varied form 0.06 grams to 3 grams. 49% of the participants used a sample intake less than 0.5 grams, 44% used 0.5 grams and 7% used more than 0.5 grams.

For the elements present in the sample above the limit of detection the calculated reproducibility is in agreement with the requirements of the target reproducibility, therefore no separate statistical analysis has been performed.

5 DISCUSSION

All of the reporting participants were able to identify the elements Cadmium, Copper and Lead in the textile sample.

When the results of this interlaboratory study were compared to the OEKO-TEX® 100 (see table below), it was noticed that not all participants would have made identical decisions about the acceptability of sample #23745.
Almost all reporting participants, except one, would have rejected the sample for Cadmium.
All reporting participants would have rejected the sample for Lead.

	Class 1: baby clothes in mg/kg	Class 2: direct skin contact in mg/kg	Class 3: no direct skin contact in mg/kg	Class 4: decoration material in mg/kg
Arsenic as As, total	100	100	100	100
Cadmium as Cd, total	40.0	40.0	40.0	40.0
Lead as Pb, total	90.0	90.0	90.0	90.0
Mercury as Hg, total	0.5	0.5	0.5	0.5

Table 6: Overview from OEKO-TEX® 100

6 CONCLUSION

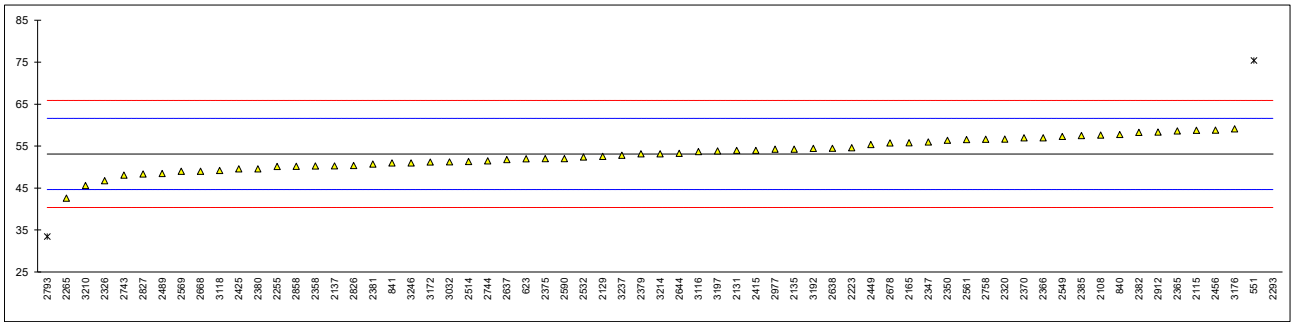
Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

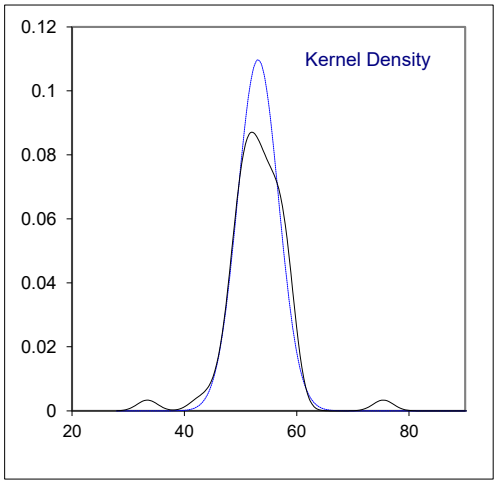
APPENDIX 1

Determination of Cadmium as Cd, total on sample #23745; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210		-----		-----	
551	EN16711-1	75.4150	R(0.01)	5.24	
623	EN16711-1	51.98		-0.27	
840	In house	57.76		1.09	
841	EN16711-1	51		-0.50	
2108	EN16711-1	57.63		1.06	
2115	EN16711-1	58.79		1.33	
2121		-----		-----	
2129	EN16711-1	52.54		-0.14	
2131	In house	54		0.21	
2135	EN16711-1	54.28		0.27	
2137	IEC62321-5	50.3		-0.66	
2165	EN16711-1	55.8		0.63	
2223	In house	54.64		0.36	
2255	EN16711-1	50.16		-0.70	
2265	EN16711-1	42.6		-2.48	
2293	EN16711-1	190.7	C,R(0.01)	32.37	first reported 70.169
2320	EN16711-1	56.671		0.83	
2326	EN16711-1	46.759		-1.50	
2347	EN16711-1	56		0.68	
2350	EN16711-1	56.40		0.77	
2358	EN16711-1	50.24		-0.68	
2365	EN16711-1	58.62		1.29	
2366	GB/T30157	57		0.91	
2370	EPA3052	57.0		0.91	
2375	EN16711-1	52		-0.26	
2379	EN16711-1	53.18		0.01	
2380	EN16711-1	49.573		-0.84	
2381	EN16711-1	50.70		-0.57	
2382	EN16711-1	58.3		1.22	
2385	EPA3052	57.5		1.03	
2415	EN16711-1	54.0		0.21	
2425	EN16711-1	49.57		-0.84	
2449	EN16711-1	55.381		0.53	
2453		-----		-----	
2456	EN16711-1	58.82		1.34	
2489	EN16711-1	48.5		-1.09	
2514	EN16711-1	51.35		-0.42	
2532	EN16711-1	52.4		-0.17	
2549	EN16711-1	57.29		0.98	
2561	EN16711-1	56.574		0.81	
2569	EN16711-1	49		-0.97	
2582		-----		-----	
2590	EN16711-1	52.002		-0.26	
2637	EN16711-1	51.8		-0.31	
2638	EN16711-1	54.47		0.32	
2644	EN16711-1	53.3		0.04	
2668	EN16711-1	49.0		-0.97	
2678	EN16711-1	55.74		0.62	
2743	EN16711-1	48.110		-1.18	
2744	EN16711-1	51.50		-0.38	
2758	EN16711-1	56.647		0.83	
2793	EN16711-1	33.42	C,R(0.01)	-4.64	first reported 67.4879
2826	EN16711-1	50.406		-0.64	
2827	EN16711-1	48.34		-1.13	
2858	EN16711-1	50.18		-0.69	
2912	EN16711-1	58.356		1.23	
2977	ISO17025-2	54.259		0.27	
2989		-----		-----	
3032	EN16711-1	51.23		-0.45	
3116	EN16711-1	53.7		0.14	
3118	EN16711-1	49.175	C	-0.93	first reported 36.656
3172	EN16711-1	51.2		-0.45	
3176	EN16711-1	59.11		1.41	
3192	EN16711-1	54.46		0.31	
3197	GB/T30157	53.86		0.17	
3210	In house	45.6		-1.77	
3214	EN16711-1	53.18		0.01	
3237	EN16711-1	52.81		-0.07	
3246	EN16711-1	51.0		-0.50	

normality	OK	
n	62	
outliers	3	
mean (n)	53.1249	
st.dev. (n)	3.63690	RSD = 7%
R(calc.)	10.1833	
st.dev.(EN16711-1:15)	4.24999	
R(EN16711-1:15)	11.9000	

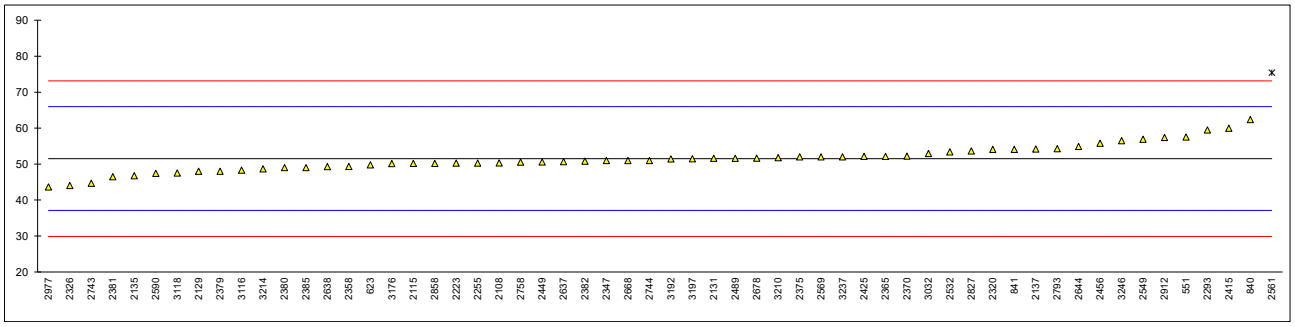


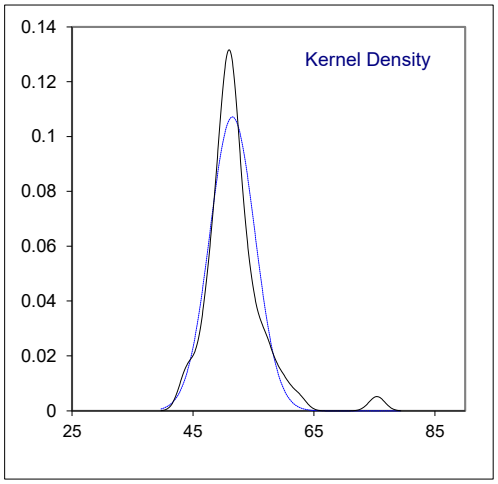


Determination of Copper as Cu, total on sample #23745; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210		-----		-----	
551	EN16711-1	57.5448		0.84	
623	EN16711-1	49.80		-0.24	
840	In house	62.40		1.51	
841	EN16711-1	54.1		0.36	
2108	EN16711-1	50.32		-0.17	
2115	EN16711-1	50.18		-0.19	
2121		-----		-----	
2129	EN16711-1	48.020		-0.49	
2131	In house	51.6		0.01	
2135	EN16711-1	46.73		-0.66	
2137	IEC62321-5	54.2		0.37	
2165		-----		-----	
2223	In house	50.27		-0.17	
2255	EN16711-1	50.28		-0.17	
2265		-----		-----	
2293	EN16711-1	59.49		1.10	
2320	EN16711-1	54.094		0.36	
2326	EN16711-1	44.033		-1.04	
2347	EN16711-1	51		-0.07	
2350	EN16711-1	not applicable		-----	
2358	EN16711-1	49.34		-0.30	
2365	EN16711-1	52.15		0.09	
2366	GB/T30157	out cap		-----	
2370	EPA3052	52.2		0.09	
2375	EN16711-1	52		0.07	
2379	EN16711-1	48.02		-0.49	
2380	EN16711-1	49.00		-0.35	
2381	EN16711-1	46.50		-0.70	
2382	EN16711-1	50.8		-0.10	
2385	EPA3052	49		-0.35	
2415	EN16711-1	60.0		1.18	
2425	EN16711-1	52.13		0.08	
2449	EN16711-1	50.57		-0.13	
2453		-----		-----	
2456	EN16711-1	55.8		0.59	
2489	EN16711-1	51.6		0.01	
2514	EN16711-1	Not Analyzed		-----	
2532	EN16711-1	53.4		0.26	
2549	EN16711-1	56.92		0.75	
2561	EN16711-1	75.424	R(0.01)	3.31	
2569	EN16711-1	52		0.07	
2582		-----		-----	
2590	EN16711-1	47.443		-0.57	
2637	EN16711-1	50.7		-0.11	
2638	EN16711-1	49.306		-0.31	
2644	EN16711-1	54.9		0.47	
2668	EN16711-1	51.0		-0.07	
2678	EN16711-1	51.64		0.02	
2743	EN16711-1	44.680		-0.95	
2744	EN16711-1	51.00		-0.07	
2758	EN16711-1	50.523		-0.14	
2793	EN16711-1	54.2862		0.38	
2826		-----		-----	
2827	EN16711-1	53.64		0.29	
2858	EN16711-1	50.22		-0.18	
2912	EN16711-1	57.383		0.81	
2977	ISO17025-2	43.670		-1.09	
2989		-----		-----	
3032	EN16711-1	52.97		0.20	
3116	EN16711-1	48.3		-0.45	
3118	EN16711-1	47.515	C	-0.56	first reported 29.538
3172		-----		-----	
3176	EN16711-1	50.14		-0.19	
3192	EN16711-1	51.44		-0.01	
3197	EN16711-1	51.48		-0.01	
3210	In house	51.8		0.04	
3214	EN16711-1	48.69		-0.39	
3237	EN16711-1	52.02		0.07	
3246	EN16711-1	56.50		0.69	

normality	OK	
n	57	
outliers	1	
mean (n)	51.5217	
st.dev. (n)	3.72198	RSD = 7%
R(calc.)	10.4215	
st.dev.(EN16711-1:15)	7.21304	
R(EN16711-1:15)	20.1965	

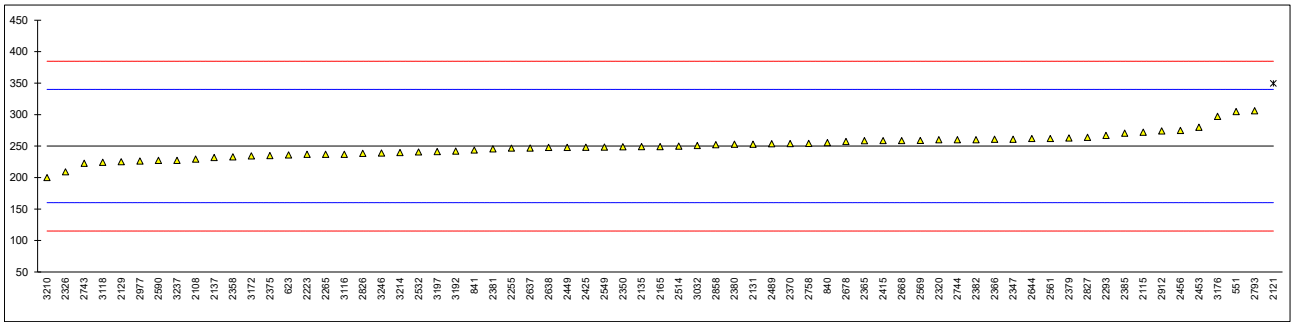


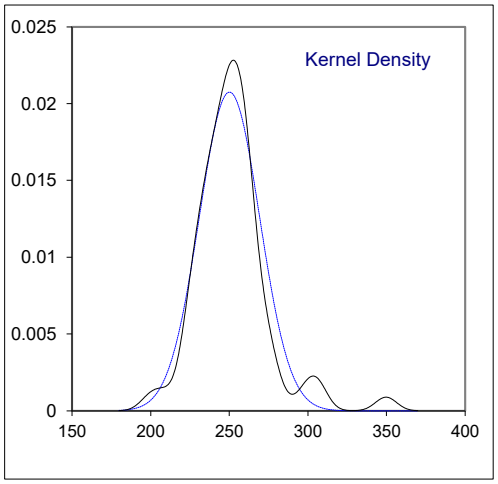


Determination of Lead as Pb, total on sample #23745; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210		-----		-----	
551	EN16711-1	304.8110		1.22	
623	EN16711-1	235.87		-0.32	
840	In house	255.39		0.12	
841	EN16711-1	243.7		-0.14	
2108	EN16711-1	229.4		-0.46	
2115	EN16711-1	272.08		0.49	
2121	CPSC-CH-E1002-08.3	349.68	R(0.01)	2.21	
2129	EN16711-1	225.06		-0.56	
2131	In house	253.2		0.07	
2135	EN16711-1	249.05		-0.02	
2137	IEC62321-5	232.0		-0.40	
2165	EN16711-1	249.1		-0.02	
2223	In house	236.86		-0.29	
2255	EN16711-1	246.74		-0.07	
2265	EN16711-1	236.9		-0.29	
2293	EN16711-1	267.08		0.38	
2320	EN16711-1	260.098		0.22	
2326	EN16711-1	209.201		-0.91	
2347	EN16711-1	261		0.24	
2350	EN16711-1	248.90		-0.03	
2358	EN16711-1	232.80		-0.38	
2365	EN16711-1	258.49		0.19	
2366	GB/T30157	261		0.24	
2370	EPA3052	254		0.09	
2375	EN16711-1	235		-0.34	
2379	EN16711-1	262.96		0.29	
2380	EN16711-1	253.139		0.07	
2381	EN16711-1	245.60		-0.10	
2382	EN16711-1	260.2		0.22	
2385	EPA3052	270.5		0.45	
2415	EN16711-1	258.7		0.19	
2425	EN16711-1	248.25		-0.04	
2449	EN16711-1	247.908		-0.05	
2453	CPSC-CH-E1002-08.3	279.85		0.66	
2456	EN16711-1	275.0		0.55	
2489	EN16711-1	253.9		0.08	
2514	EN16711-1	250.06		0.00	
2532	EN16711-1	240.7		-0.21	
2549	EN16711-1	248.41		-0.04	
2561	EN16711-1	262.12		0.27	
2569	EN16711-1	259		0.20	
2582		-----		-----	
2590	EN16711-1	227.284		-0.51	
2637	EN16711-1	247		-0.07	
2638	EN16711-1	247.9		-0.05	
2644	EN16711-1	262		0.26	
2668	EN16711-1	258.9		0.20	
2678	EN16711-1	257.44		0.16	
2743	EN16711-1	222.840		-0.61	
2744	EN16711-1	260.10		0.22	
2758	EN16711-1	254.247		0.09	
2793	EN16711-1	306.2260		1.25	
2826	EN16711-1	238.596		-0.26	
2827	EN16711-1	264.01		0.31	
2858	EN16711-1	252.24		0.05	
2912	EN16711-1	274.18		0.53	
2977	ISO17025-2	226.338		-0.53	
2989		-----		-----	
3032	EN16711-1	250.99		0.02	
3116	EN16711-1	237		-0.29	
3118	EN16711-1	224.140		-0.58	
3172	EN16711-1	234.6		-0.34	
3176	EN16711-1	297.24		1.05	
3192	EN16711-1	242.17		-0.18	
3197	In house	241.38		-0.19	
3210	In house	200		-1.11	
3214	EN16711-1	239.64		-0.23	
3237	EN16711-1	227.39		-0.50	
3246	EN16711-1	239.11		-0.24	

normality	suspect	
n	66	
outliers	1	
mean (n)	250.1059	
st.dev. (n)	19.23258	RSD = 8%
R(calc.)	53.8512	
st.dev.(EN16711-1:15)	45.01906	
R(EN16711-1:15)	126.0534	





APPENDIX 2 Other reported elements

Other reported elements on sample #23745; results in mg/kg

lab	Sb	As	Cr	Co	Hg	Ni
210	-----	-----	-----	-----	-----	-----
551	0.0394	0.0900	1.9625	0.0088	0.0000	1.7518
623	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
840	<10	<10	<10	<10	<10	<10
841	<10	<10	<10	<10	<10	<10
2108	not detected	not detected	not detected	not detected	not detected	not detected
2115	-----	-----	1.14	-----	-----	0.77
2121	-----	-----	-----	-----	-----	-----
2129	not detectable	not detectable	not detectable	not detectable	not detectable	not detectable
2131	not detected	not detected	not detected	not detected	not detected	0.9
2135	-----	-----	1.25	-----	-----	1.17
2137	-----	-----	-----	-----	-----	-----
2165	Not Detected	-----	Not Detected	-----	Not Detected	-----
2223	<0.01	<0.01	1.06	<0.01	0.00	0.74
2255	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2265	-----	not detected	not detected	-----	not detected	-----
2293	0	0.039	1.28	0	0.0778	0.835
2320	<5	<5	<5	<5	<0.1	<5
2326	ND	ND	ND	ND	ND	ND
2347	<10	<10	<10	<10	<10	<10
2350	<10	<10	not applicable	<5	<0.1	not applicable
2358	not detected	not detected	not detected	not detected	not detected	not detected
2365	＜10	＜10	＜10	＜10	＜10	＜10
2366	-----	-----	-----	-----	-----	-----
2370	<2	<2	<2	<2	<2	<2
2375	<10	<10	<10	<10	<10	<10
2379	<2	<2	<2	<2	<0.1	<2
2380	-----	-----	-----	-----	-----	-----
2381	-----	-----	-----	-----	-----	-----
2382	not detected	not detected	not detected	not detected	not detected	not detected
2385	<5	<2	<5	<1	<0.1	<5
2415	-----	-----	-----	-----	-----	-----
2425	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2449	-----	-----	-----	-----	-----	-----
2453	-----	-----	-----	-----	-----	-----
2456	not detected	not detected	1.28	not detected	not detected	not detected
2489	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2514	Not Tested	Not Tested	Not Analyzed	Not Analyzed	Not Analyzed	Not Analyzed
2532	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2549	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2561	-----	-----	-----	-----	-----	-----
2569	NOT DETECTED	NOT DETECTED	NOT DETECTED	NOT DETECTED	NOT DETECTED	NOT DETECTED
2582	-----	-----	-----	-----	-----	-----
2590	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.	< L.O.Q.
2637	<0.02	<0.01	1.06	<0.005	<0.005	0.68
2638	not detected	not detected	1.07	not detected	not analysed	1.31
2644	not detected	not detected	not detected	not detected	not detected	not detected
2668	-----	-----	-----	-----	-----	-----
2678	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2743	-----	-----	-----	-----	-----	-----
2744	Not Detected	Not Detected	0.95	Not Detected	Not Detected	0.80
2758	not detected	not detected	1.168	not detected	not detected	1.084
2793	not detected	not detected	not detected	not detected	not detected	not detected
2826	-----	Not detected	-----	-----	Not detected	-----
2827	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2858	not detected	not detected	not detected	not detected	not detected	not detected
2912	<0.10	<0.10	1.139	<0.10	<0.01	<0.10
2977	not detected	not detected	not detected	not detected	not detected	not detected
2989	-----	-----	-----	-----	-----	-----
3032	not detected	not detected	not detected	not detected	not detected	not detected
3116	<5	<5	<5	<5	<5	<5
3118	<5	<2.5	<5	<5	<5	<5
3172	< 10	< 10	< 10	< 10	< 10	< 10
3176	-----	-----	1.10	-----	-----	0.69
3192	0.11	<0,01	1.14	<0,01	<0,01	0.67
3197	<2	<2	<2	<2	<2	<2
3210	<25	<25	<10	<10	<0.02	<10
3214	<5	<0.5	<5	<5	<0.1	<5
3237	-----	-----	-----	-----	-----	-----
3246	nd	nd	nd	nd	nd	nd

APPENDIX 3 Analytical Details

lab	ISO/IEC1702 accr.	sample preparation	Sample intake (g)
210	---	---	
551	---	---	
623	Yes	Further cut	0.5
840	Yes	Further cut	1g
841	Yes	Further cut	0.25 grams
2108	Yes	Further cut	0,1 g
2115	Yes	Used as received	0.1 g
2121	Yes	Further cut	85mg
2129	Yes	Further cut	ca. 1g
2131	Yes	Used as received	0.5g
2135	Yes	Used as received	0,3g
2137	Yes	Further cut	0.1
2165	Yes	Further cut	0.1g, nearest 0.1mg
2223	Yes	Further cut	2
2255	Yes	Used as received	0.2148
2265	Yes	Further cut	0,1 grams
2293	Yes	Further cut	0.2 g
2320	Yes	Further cut	0.5
2326	Yes	Further cut	0.5 GM
2347	Yes	Further cut	0.2g
2350	Yes	Further cut	approximately 0.5g
2358	Yes	Further cut	0.5
2365	Yes	Used as received	0.2g
2366	Yes	Further cut	
2370	Yes	Used as received	0.5g
2375	Yes	Further cut	0,1 gram
2379	Yes	Further cut	0.5 g
2380	Yes	Further cut	0.5 g
2381	Yes	Further cut	0.25g
2382	Yes	Further cut	0.2g
2385	Yes	Further cut	0.1 to 0.2
2415	No	Used as received	0.1 gram
2425	Yes	Further cut	0.5g
2449	Yes	Further grinded	0.1
2453	Yes	Used as received	±0.2g
2456	Yes (not for Hg)	Further cut	
2489	Yes	Further cut	0.1036g
2514	Yes	Used as received	0.1630
2532	Yes	Further cut	0.2 grams
2549	Yes	Used as received	0.5 grams
2561	Yes	Used as received	0.06
2569	Yes	Further cut	0.5 gm
2582	---	---	
2590	Yes	Used as received	0.2 g
2637	Yes	Further cut	0.5 g
2638	No	Further cut	0.5 gm
2644	Yes	Used as received	0.5
2668	Yes	Further cut	0.5535 g
2678	No	Used as received	0.5 g
2743	Yes	Used as received	0.2g
2744	Yes	Used as received	0,5
2758	No	Used as received	0.5
2793	Yes	Used as received	0.5015g
2826	Yes	Further cut	0.5012g
2827	Yes	Further cut	0.5g
2858	Yes	Further cut	0.5 gm
2912	Yes	Used as received	
2977	---	---	
2989	---	---	
3032	Yes	Further cut	0.202 grams
3116	Yes	Used as received	0.5
3118	Yes	Further cut	3 grams
3172	Yes	---	
3176	Yes	Further cut	0.5
3192	Yes	Used as received	0,5 g
3197	Yes	Further cut	0.5 g
3210	Yes	Further cut	0.1005
3214	Yes	Used as received	0.15g
3237	Yes	Used as received	0,1
3246	Yes	Further cut	0.2g

APPENDIX 4

Number of participants per country

6 labs in BANGLADESH
1 lab in BRAZIL
2 labs in FRANCE
7 labs in GERMANY
1 lab in GUATEMALA
3 labs in HONG KONG
7 labs in INDIA
2 labs in INDONESIA
8 labs in ITALY
2 labs in KOREA, Republic of
1 lab in MOROCCO
5 labs in P.R. of CHINA
4 labs in PAKISTAN
1 lab in PORTUGAL
2 labs in SRI LANKA
3 labs in SWITZERLAND
2 labs in TAIWAN
1 lab in THAILAND
2 labs in TUNISIA
5 labs in TURKEY
1 lab in UNITED KINGDOM
4 labs in VIETNAM

APPENDIX 5

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?

Literature

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